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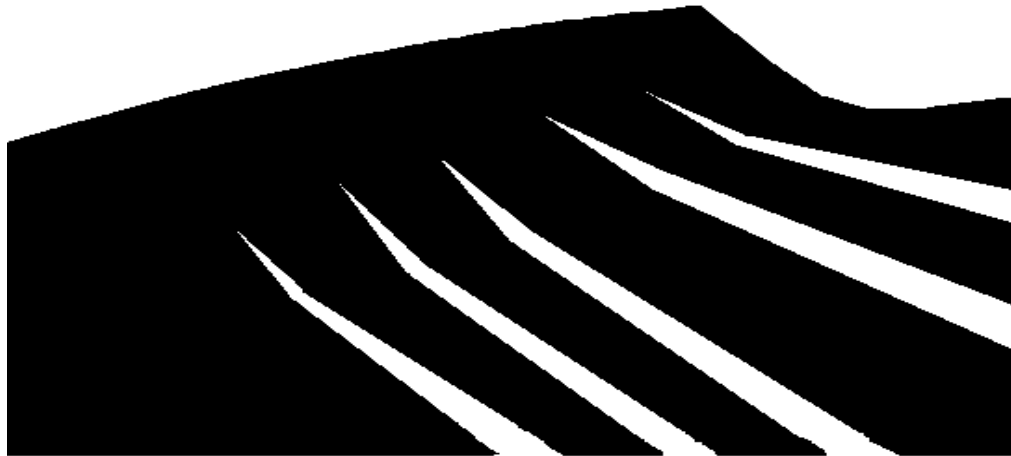
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LANL-CST-DP-15, R4

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CRUSHED ROCK COLUMN STUDIES

LOS ALAMOS QUALITY PROGRAM



APPROVAL FOR RELEASE

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Los Alamos

Yucca Mountain Site

Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	03/05/80	N/A	Not applicable.
R1	08/30/82	N/A	Revision 0 of this procedure was previously identified as TWS-CNC11-DP-15.
R2	04/03/89	N/A	Revision 1 of this procedure was previously identified as TWS-CNC-DP-15.
R3	08/13/92	All	Complete rewrite. Revision 2 of this procedure was previously identified as TWS-INC-DP-15
R4	03/21/97	All	Revised to comply with LANL-YMP-QP-06.3 requirements. Revision 3 of this procedure was previously identified as LANL-INC-DP-15.

Los AlamosYucca Mountain Site
Characterization Project

CRUSHED ROCK COLUMN STUDIES

1.0 PURPOSE

This detailed technical procedure (DP) describes dynamic transport tests on crushed rock columns by studying the flow of a known tracer with a known flow rate through a column of crushed rock for the Yucca Mountain Site Characterization Project (YMP).

2.0 SCOPE

This DP applies to all YMP personnel who assemble, prepare, and sample crushed rock columns within the Dynamic Transport Task of the Los Alamos National Laboratory (Los Alamos) YMP.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-CST-DP-35, pH Measurements
LANL-CST-DP-62, Bulk NTS Well Water Samples
LANL-CST-DP-63, Preparation of NTS Core Samples for Crushed Rock Experiments
CST-SOP-037, Rules for Handling Radioactive Material at TA-48

4.0 DEFINITIONS

4.1 Tracer Solution

The tracer solution is the solution containing the substance(s) whose diffusion behavior is to be studied.

4.2 Eluate

The eluate is the solution resulting from an elution process.

4.3 Collection Time

The collection time is the time interval between collection of eluate samples.

4.4 Flow Rate

The flow rate is the amount of liquid eluted through the column per unit of time.

4.5 $c_o(t)$

The tracer concentration in initial tracer solution in time (i.e., the aliquot of the initial tracer solution taken at the beginning of the experiment) corresponds to $C_o(t=0)$.

4.6 $c_i(t)$

The tracer concentration in solution I at time t (solution I represents the eluate collected during them t_i).

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- Principal Investigator (PI)
- Procedure Users

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or the current work practice is documented in accordance with QP-03.5, subsection 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

6.1 Principal

This procedure describes the set up and proper conduct of crushed rock column experiments. Results are used to evaluate batch sorption measurements under flowing conditions. By comparing differences between batch measurements and crushed rock column measurements, one will be able to study multiple species formation, colloid formation, and other geochemical reactions.

6.2 Equipment and Hardware/Software

Equipment needed is listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the user or the PI.

- syringe pump
- calibrated balance
- commercially available measuring tools (i.e., tape measure, ruler, micrometer, or caliper)
- injection valve
- Polyether Etherketone (PEEK) or teflon tubing
- Polyethylene frits and Luer fittings
- fraction collector vials
- test tubes

6.2.1 Equipment Malfunctions

The equipment needed to conduct this experiment must be clean and in good operating order. Do not exceed the capacity of the syringe pumps (generally ~35 psi). The computerized syringe pumps are susceptible to surges, spikes and general power failure such that they instantly turn themselves off, in this event the pumps must be turned back on.

6.2.2 Safety Considerations

Ensure compliance with CST Division Environmental Safety and Health Operational Policy Statement.

6.2.3 Special Handling

Tracer Solutions for these experiments are often radioactive and should be handled in accordance with the Rules for Handling Radioactive Material at TA-48 (CST-SOP-037). Injection of radionuclides often leaves residue sorbed to minerals comprising the crushed rock material of the column. The crushed rock material, after injection, should be handled as potentially contaminated and if to be disposed, handled as low level waste.

6.3 Preparatory Verification

There are no critical setup parameters, prerequisites, or mandatory verification points.

6.3.1 Hold Points

There are no hold points for this procedure

6.3.2 Calibration

6.3.2.1 Balances used for weighing are required to be calibrated pursuant to QP-12.3. When data are collected from a balance, the unique identifier number of that balance is recorded in the user's laboratory notebook along with the data collected.

6.3.2.2 All pH determinations shall be conducted in accordance with DP-35. The unique identifier of the pH meter shall be recorded in the user's laboratory notebook.

6.3.2.3 The measuring instruments used (i.e., tape measure, ruler, caliper, or micrometer) are commercial-grade and do not require calibration pursuant to QP-12.3.

6.3.3 Environmental Conditions

No special environmental conditions are required for this DP. If any special conditions are used, they will be recorded according to subsection 6.7 of this DP.

6.4 Control of Samples

All samples will be controlled according to QP-08.1.

6.5 Implementing Procedure

6.5.1 Prepare the crushed rock according to directions in DP-63.

6.5.2 Prepare the column.

6.5.2.1 Make the column from acrylic or Teflon tubing with end caps machined from polyether Etherketone (PEEK) material. Polyethylene frits are utilized in the end caps to hold in the material. Refer to Attachment 1 for correct assembly of the column.

6.5.2.2 Measure the length of the column to within 0.25 in., using a tape measure or ruler. Record value in notebook.

6.5.2.3 Weigh the empty column. Record value in notebook.

6.5.3 Prepare the Tracer Solution

6.5.3.1 Prepare a solution of the appropriate ground water containing the tracer(s) to be used. The source of the ground water and tracer(s) are specified by the PI. Record the type of tracer and the method of preparation in the notebook.

6.5.3.2 Measure the pH of the solution according to DP-35 and record in the notebook.

6.5.4 Pack the Column with either Dry or Wet Crushed Rock

6.5.4.1 For a dry packed column, pour the rock into the column through the funnel. If necessary, tap or vibrate column to make the rock reach the bottom of the column. Some banding may appear as a result of differences in particle densities; small, numerous bands are desirable. If the banding is too wide, remove the rock from the column. Finally remove the funnel and replace it with an end cap and frit. If settling occurs when water is pumped through the column, remove the top Luer fitting and frit, replace the funnel, and add more crushed rock. Weigh the packed column and record weight in the notebook.

6.5.4.2 For a wet packed column, pour a slurry of crushed rock and water into the column via a funnel. When the column is full, remove the funnel and attach an end cap and frit. If settling occurs, remove the top end cap and frit, replace the funnel, and add more crushed rock. Weigh the packed column and record value in the notebook.

6.5.5 Prepare the pump by filling it with the appropriate ground water, as specified by the PI. Check all the connections from the pump to the injection valve (i.e., the injection valve loop, from the injection valve to the column, and from the column to the collection vessel.

6.5.6 Place the pre-weighed vials to be used for eluate collection in the fraction collector. Check injection vessel or collection vials for correct loading in the fraction collector. Record weights and vials in sequence.

6.5.7 Ensure that the following data are recorded in a notebook:

- Unique identifier of the sample used to prepare the column (carried from the procedure DP-63). If more than one column has been prepared from the same NTS sample, an additional alphanumeric character will be added to the identifier in order to maintain uniqueness. The length and diameter of the crushed rock column.
- The weight of empty column.
- The weight of packed column, dry packed or wet packed.
- The unique identifier of the water used.
- The tracer solution to be used and analytical method of preparation (e.g., the experimental steps used for preparation, notebook number and pages used specifying these steps, or a detailed procedure).

- The unique identifier of analytical balance used.
- The flow rate through the crushed rock column.
- The volume of tubing from syringe or valve to column.
- The volume of tubing from column to collection vessel.

6.5.8 Conduct the Experiment

- 6.5.8.1 Load the traced solution in the injection valve loop using a syringe. Make sure the injection valve is turned to the *load* position.
- 6.5.8.2 After the contents of the syringe has been loaded, turn the injection valve to *inject* and turn on the fraction collector for vial collection or insert the tube into the collection vessel.
- 6.5.8.3 Record the date and time.
- 6.5.8.4 Place an aliquot of the tracer solution in a container for analysis (the tracer concentration of this aliquot will be used to determine C_0 at an arbitrary time t).
- 6.5.8.5 Ensure that the following entries are recorded in a laboratory notebook:
- The volume of the injection loop.
 - The start time of eluate collection and amount of initial tracer solution to be analyzed, specifying the units of volume or weight used for measuring amount.
 - The start time of the injection.
 - The tare weight and total weight of each vial.
 - The ID# of analytical balance used.
 - The analytical technique used and a reference to the detailed procedure used for this technique.
 - The collection time to be used on fraction collector.
- 6.5.8.6 At the conclusion of the experiment (determined by the PI based on the analytical results of the periodic samples), determine the pH (according to DP-35) of the solution in the non-tracer reservoir and record the data in the notebook.

6.6 Data Acquisition and Reduction

The tare and the final weight of eluate vials are later reduced by subtracting the tare weights from final weights of the collection vials and dividing by the elapsed time in order to obtain the flow rate of the column. The relative concentration of the tracer, $C_i(t)/C_o(t)$, will be calculated for each eluate collected.

6.7 Potential Sources of Error and Uncertainty

The most common source of error that results in data rejection is leakage of the column. Leakage can be caused by loose Luer connectors. Leakage is detected by periodic visual inspection. If leakage is detected, record this problem in the laboratory notebook and reject the data.

Evaporation of the tracer in the collection vials can cause errors in the results. Consequently, keep the collection vials capped after eluate collection.

7.0 RECORDS

Records resulting from the proper execution of this DP are entries in the laboratory notebooks and QA approved software. Electronic data shall be documented in accordance with QP-03.5.

8.0 ACCEPTANCE CRITERIA

Proper recording of the data specified in subsections 6.5.8 and 6.5.10.6 constitute the acceptance criteria for this DP.

9.0 TRAINING

The PI or his designee will formally train the users assigned to implement this DP. The training will require observation and evaluation of the performance of the trainee as he follows this DP. Training will be documented in accordance with QP-02.7.

10.0 ATTACHMENTS

Attachment 1: Cross-Sectional View of Crushed Rock Column (1 page)

